

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
Reply to Office Action of February 18, 2009

••• R E M A R K S / A R G U M E N T S •••

The Official Action of February 18, 2009 has been thoroughly studied. Accordingly, the following remarks are believed to be sufficient to place the application into condition for allowance.

Claims 1-8 are pending in this application.

On page 3 of the Office Action the Examiner has rejected claims 1-8 under 35 U.S.C. §112, second paragraph as failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Under this rejection the Examiner has taken the position that the recitation "general formula" is unclear.

In response to this basis for rejecting the claims, the undersigned conducted a search on the phrase "general formula" in the "Claims" field on the U.S. Patent Website data base. As a result 37131 patents were identified as having the phrase "general formula" in the claims.

This indicates that the phrase "general formula" is well known and understood by those skilled in the art.

A further search on the U.S. Patent Website data base indicates that the Examiner has issued a number of patents with the phrase "general formula" in the claims.

Based upon the above, it is unclear why the Examiner is rejecting applicants' claims because of the recitation of "general formula."

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
Reply to Office Action of February 18, 2009

On page 4 of the Office Action the Examiner has rejected claims 1-8 under 35 U.S.C. §112, first paragraph as containing subject matter which was not described in the specification in such a way to reasonably convey to one skilled in the art that the inventors, at the time the application was filed, had possession of the claimed invention.

Under this rejection the Examiner has taken the position that:

1. The recitation of "paraformaldehyde" in claim 1 is considered new matter; and
2. The process of new claim 5 is new matter, because there is no process in the specification

which allows reaction with trioxane.

"Paraformaldehyde" is discussed in applicants' paragraph [0008] and Example 1 (paragraph [0014]).

The disclosure of a reaction with trioxane is discussed in applicants' paragraph [0008]

It is accordingly submitted that applicants' disclosure satisfies the requirements of 35 U.S.C.

§112, first and second paragraphs.

Claims 1-4 and 6-8 stand rejected under 35 U.S.C. §103(a) as being unpatentable over Weinmayr (JOC, 28, 492-4, 1963) JP 61-097277 to Yonosuke et al. ("Yonosuke '277") and JP 63-146868 to Yonosuke et al. ("Yonosuke '868").

The Examiner has relied upon Weinmayr as teaching:

...a process for producing 2,2,3,3-tetrafluorooxethane by reacting fluoro-olefins with formaldehyde in hydrogen fluoride. Trifluoroethylene condenses with formaldehyde in liquid hydrogen fluoride to form 2,2,3,3-pentafluoro-1-propanol and fluoromethyl 1-2,2,3,3,-pentafluoropropyl ether. See the entire document especially abstract, 1-32, 1<sup>st</sup> column on page 493.

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
Reply to Office Action of February 18, 2009

The Examiner has relied upon Yonosuke '277 as teaching 2,2,3,3-tetrafluorooxethane.

The Examiner has also relied upon Yonosuke '868 as teaching 2,2,3,3-tetrafluorooxethane.

The Examiner states that:

[Applicants'] Presently claimed invention is drawn to a process for producing 2,2,3,3-tetrafluorooxethane, which comprises allowing tetrafluoroethylene to react with a formaldehyde compound in anhydrous hydrogen fluoride reaction being carried out in the presence of polyfluoroalkylic acid or polyfluroralky' ester thereof, represented by the formula: RfCOORf".

The Examiner states that:

Instant claims for process for producing 2,2,3,3-tetrafluorooxethane has been generically taught by the reference.

The Examiner takes the position that:

It would have been obvious....to prepare 2,2,3,3-tetrafluorooxethane, by formaldehyde or its derivative and anhydrous hydrogen fluoride because prior art teaches this method. JP references also teach 2,2,3,3-tetrafluorooxethane (since there is criticality of invention has been disclosed in the specification examiner considered that claims are obvious over the prior art. Final decision will be made after considering the translation of JP REFERENCES. The reaction to prepare 2,2,3,3-tetrafluorooxethane and its derivatives has been taught by the reference.

Weinmayr teaches that the 2,2,3,3-tetrafluorooxethane is formed as a "minor by-product." In this regard, Weinmayr is concerned with forming 2,2,3,3,3-pentafluoro-1-propanol and fluoromethyl-2,2,3,3,3-pentafluoropropyl ether.

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
Reply to Office Action of February 18, 2009

On page 492, right hand column, lines 19-25, Weinmayr discloses that "small quantities of 2,2,3,3 [where 2,2,3,3,3 must be a clerical error]-tetrafluorooxetane  $CF_2CF_2CH_2O$ , b.p. 28°C – are also formed."

Furthermore, on page 492, right hand column, lines 7-55, Weinmayr discloses that "Upon careful fractionation 300 g of 2,2,3,3-tetrafluorooxetane, b.p. 28°C, and....were obtained;" however, due to the indefinite disclosure such as "Nine condensations made as described above were combined..." it is not possible to calculate the percentage yield of the 2,2,3,3- tetrafluorooxetane obtained by Weinmayr.

The Examiner had previously cited Weinmayr at "1-32 1<sup>st</sup> column on page 493."

Applicants note that on page 493, left hand column, lines 1-32 that there is no disclosure of 2,2,3,3- tetrafluorooxetane.

On page 493, in the right hand column under "Reaction of Tetrafluoroethylene with Formaldehyde," Weinmayr discloses "6 moles of formaldehyde is made to react with 3 moles of tetrafluoroethylene [TFE] in the presence of 600 g of HF to synthesize 429 (2.5 moles) of 2,2,3,3,3-pentafluoro-1-propanol in yield of 8.7% in terms of TFE, wherein 61 g (0.34 moles) of fluoromethyl-2,2,3,3,3-pentafluoropropyl ether is obtained as a by-product in yield of 11.3% in terms of TFE; total yield being 96.0%."

This means that the reaction is mainly directed to formation of 2,2,3,3,3-pentafluoro-1-propanol, where 2,2,3,3-tetrafluorooxetane, when formed as a by-product, has a yield of 4.0% at the most.

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
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In applicants' specification there is presented a Comparative Example that shows a process as disclosed by in JOC by Weinmayr where the yield of 2,2,3,3-tetrafluoroethane was 18.8%. (See page 6 of applicants' specification)

In contrast, according to the present invention which is based upon the reaction in the presence of polyfluoroalkyl carboxylic acid or a polyfluoroalkyl ester, the yield of 2,2,3,3-tetrafluoroethane is 35.3 to 39.3%, which is almost double as shown in the results of applicants' Examples 1-4.

It is submitted that applicants' claimed process for producing 2,2,3,3-tetrafluoroethane (as a product as opposed to a minor by-product in the case of Weinmayr) is not at all taught or suggested by Weinmayr.

Applicants' process produces yields of 2,2,3,3-tetrafluoroethane that are significantly greater than Weinmayr.

Accordingly, it would **not** have been obvious...."to prepare 2,2,3,3-tetrafluoroethane, by formaldehyde or its derivative and anhydrous hydrogen fluoride" as the Examiner concludes. Rather, it is submitted that applicants' results are **unexpected** over the teachings of Weinmayr.

If anything, it would go against the teachings of Weinmayr to make or propose any modification that would increase the yield of by-products to the detriment of producing a greater desired product yield.

It is submitted that such an improper modification is tantamount to destroying or at least departing from the teachings of Weinmayr.

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
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An English translation of each of Yonosuke '277 is being submitted herewith for the Examiner's review. Also submitted is EP 0 252 454 which corresponds to Yonosuke '868 (See applicants' International Search Report).

Yonosuke '277 and Yonosuke '868 neither teach nor suggest a reaction that is carried out in the presence of polyfluorocarboxylic acid or polyfluoroalkyl ester with tetrafluoroethylene being reacted with a compound that generates formaldehyde in anhydrous hydrogen fluoride to form 2,2,3,3-tetrafluoroethane.

Therefore, the teachings of Yonosuke '277 and Yonosuke '868 are not actually relevant and do not cure the deficiencies of Weinmayr.

It is noted that Yonosuke '277 and Yonosuke '868 were cited as category A references in the applicants' International Search Report, because they were only considered as being of general interest.

Further in the Written Opinion of the International Searching Authority, the claims were held to be novel, involve an inventive step and have industrial applicability over Yonosuke '277 and Yonosuke '868.

Based upon the above distinctions between the prior art relied upon by the Examiner and the present invention, and the overall teachings of prior art, properly considered as a whole, it is respectfully submitted that the Examiner cannot rely upon the prior art as required under 35 U.S.C. §103 to establish a *prima facie* case of obviousness of applicants' claimed invention.

Appl. No. 10/589,418  
Amendment Dated May 5, 2009  
Reply to Office Action of February 18, 2009

It is, therefore, submitted that any reliance upon prior art would be improper inasmuch as the prior art does not remotely anticipate, teach, suggest or render obvious the present invention.

It is submitted that the claims, as now amended, and the discussion contained herein clearly show that the claimed invention is novel and neither anticipated nor obvious over the teachings of the prior art and the outstanding rejection of the claims should hence be withdrawn.

Therefore, reconsideration and withdrawal of the outstanding rejection of the claims and an early allowance of the claims is believed to be in order.

It is believed that the above represents a complete response to the Official Action and reconsideration is requested.

If upon consideration of the above, the Examiner should feel that there remain outstanding issues in the present application that could be resolved, the Examiner is invited to contact applicants' patent counsel at the telephone number given below to discuss such issues.

Please charge the any fees due in connection with the filing of this paper to Deposit Account No. 12-2136 and please credit any excess fees to such deposit account.

Respectfully submitted,



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## EUROPEAN PATENT APPLICATION

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⑭ Date of filing: 03.07.87

⑮ Priority: 03.07.86 JP 157701/86

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⑯ Removal of hydrogen fluoride from 2,2,3,3-tetrafluoroacetane.

⑰ By extracting hydrogen fluoride from 2,2,3,3-tetrafluoroacetane with a halogenated hydrocarbon, a mixture of 2,2,3,3-tetrafluoroacetane and the halogenated hydrocarbon containing a small amount of hydrogen fluoride is obtained. Said mixture may be distilled at least twice to obtain a mixture of 2,2,3,3-tetrafluoroacetane and the halogenated hydrocarbon containing substantially no hydrogen fluoride.

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0252454TITLE OF THE INVENTION

REMOVAL OF HYDROGEN FLUORIDE FROM 2,2,3,3-TETRA-  
FLUOROOXETANE

BACKGROUND OF THE INVENTIONField of the Invention

The present invention relates to removal of hydrogen fluoride from 2,2,3,3-tetrafluorooxetane (hereinafter referred to as "tetrafluorooxetane"). More particularly, it relates to a process for removing hydrogen fluoride from tetrafluorooxetane comprising extracting hydrogen fluoride from tetrafluorooxetane with a halogenated hydrocarbon and optionally at least twice distilling tetrafluorooxetane from which hydrogen fluoride is extracted with the halogenated hydrocarbon.

10 Description of the Prior Arts

Tetrafluorooxetane is useful as a solvent or a monomer for producing a straight chain polyether, and it is produced by reacting tetrafluoroethylene and paraformaldehyde in anhydrous hydrogen fluoride. However, separation of hydrogen fluoride from tetrafluorooxetane by distillation since boiling points of the former and the latter are 20°C and 28°C, respectively. Conventionally, hydrogen fluoride is removed from tetrafluorooxetane by washing tetrafluoro-oxetane containing hydrogen fluoride with water. The washing method still has some problems such as neutralization and drying of washed tetrafluorooxetane and is not suitable for industrial application.

- 2 -

0252454

SUMMARY OF THE INVENTION

One object of the present invention is to provide a process for removing hydrogen fluoride from tetrafluoro-oxetane which does not require post-treatments such as 5 neutralization and drying.

Another object of the present invention is to provide a process for removing hydrogen fluoride from tetra-fluorooxetane suitable for industrial application.

These and other object of the present invention 10 are achieved by a process for removing hydrogen fluoride from tetrafluorooxetane comprising extracting hydrogen fluoride from tetrafluorooxetane with a halogenated hydrocarbon to obtain a mixture of tetrafluorooxetane and the halogenated hydrocarbon containing a small amount of hydrogen 15 fluoride, and optionally distilling said mixture to obtain a mixture of tetrafluorooxetane and the halogenated hydrocarbon containing a decreased amount of hydrogen fluoride and distilling the mixture of tetrafluorooxetane and the halogenated hydrocarbon to recover the mixture containing 20 substantially no hydrogen fluoride.

DETAILED DESCRIPTION OF THE INVENTION

When tetrafluorooxetane produced by reacting 25 tetrafluoroethylene and paraformaldehyde in hydrogen fluoride as described is directly distilled, an azeotropic mixture containing about 70 % by weight of tetrafluorooxetane and about 30 % by weight of hydrogen fluoride. When such

- 3 -

0252454

azeotropic mixture is extracted with the halogenated hydrocarbon, a mixture of tetrafluorooxetane and the halogenated hydrocarbon containing a small amount, for example 0.1 to 1.0 % by weight, preferably 0.2 to 0.5 % by weight of hydrogen fluoride is obtained. Tetrafluorooxetane containing such small amount of hydrogen fluoride can be used for further reaction or in the final use without further purification.

Examples of the halogenated hydrocarbon used for the extraction of hydrogen fluoride from tetrafluorooxetane are trichloroethylene, tetrachloroethane, 1,1,1- or 1,1,2-trifluorotrichloroethane, 1,2-difluorotetrachloroethane, methylchloroform, tetrachloroethylene, p-chlorotoluene, p-chlorobenzotrifluoride and 3,4-dichlorobenzotrifluoride as well as mixtures thereof. Among them, 1,1,2-trichloro-1,2,2-trifluoroethane and 1,2-difluorotetrachloroethane are preferred.

According to the present invention, tetrafluoro-oxetane containing a small amount of hydrogen fluoride may be further purified by distillation. The distillation is carried out at least twice.

When 1,1,2-trichloro-1,2,2-trifluoroethane is used as the halogenated hydrocarbon, the first and second distillations may be carried out under following conditions:

- 4 -

0252454

First distillation

Pressure: Atmospheric pressure to 5 kg/cm<sup>2</sup>G,  
preferably to 2 kg/cm<sup>2</sup>G

Temperature of the bottom: 40 to 80°C

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Second distillation

Pressure: Atmospheric pressure to 5 kg/cm<sup>2</sup>G,  
preferable to 2 kg/cm<sup>2</sup>G

Temperature of the distillate: 30 to 70°C

By the first distillation, a mixture of tetra-  
10 fluorooxetane and the halogenated hydrocarbon containing  
less than 500 ppm, preferably 300 ppm of hydrogen fluoride  
is obtained as a bottom. Therefore, the bottom mixture  
obtained by the first distillation is further distilled to  
reduce the content of the halogenated hydrocarbon in tetra-  
15 fluorooxetane.

After the second distillation, an azeotropic mix-  
ture of tetrafluorooxetane and the halogenated hydrocarbon  
containing substantially no hydrogen fluoride, namely less  
than 500 ppm of hydrogen fluoride is recovered. Since the  
20 halogenated hydrocarbon is a solvent, the mixture of tetra-  
fluorooxetane and the halogenated hydrocarbon can be used  
for polymerization of tetrafluorooxetane to produce a  
straight chain polyether without further separation of them.

25 The present invention will be explained further in  
detail by following Examples.

- 5 -

0252454

Example 1

An azeotropic mixture of 70 % by weight of tetrafluorooxetane and 30 % by weight of hydrogen fluoride being supplied at a rate of 20 l/hr. was extracted by circulating 5 1,1,2-trichloro-1,2,2-trifluoroethane in the mixture at a rate of 90 l/hr. at a temperature of 0 to 5°C under pressure of 0.8 kg/cm<sup>2</sup>G. to obtain a mixture of tetrafluorooxetane and 1,1,2-trichloro-1,2,2-trifluoroethane (weight ratio of 1:9) containing 0.2 to 0.5 % by weight of hydrogen fluoride.

Example 2

The mixture obtained in Example 1 was subjected to first distillation under following conditions:

Distillation Pressure:	0.8 kg/cm <sup>2</sup> G
Supply amount of the mixture:	100 l/hr.
Temperature of the bottom:	56°C
Outlet rate of the bottom:	97 l/hr.
Composition of the bottom:	
Hydrogen fluoride:	200 ppm
Tetrafluorooxetane	6 wt.%
1,1,2-trichloro-1,2,2-trifluoroethane:	94 wt.%

20 Then, the bottom from the first distillation was again distilled under following conditions:

Distillation pressure:	0.8 kg/cm <sup>2</sup> G
Rate of the mixture received from	
the still pot in first distillation:	97 l/hr.
Running temperature:	40°C

- 6 -

**0252454**

Running rate: 10 l/hr.

By the second distillation, a mixture of tetra-fluoroacetane and 1,1,2-trichloro-1,2,2-trifluoroethane of a weight ratio of 85:15 containing 100 to 500 ppm of hydrogen fluoride was obtained.

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0252454

- 7 -

## CLAIMS:

1. A process for removing hydrogen fluoride from 2,2,3,3-tetrafluorooxetane comprising extracting hydrogen fluoride from 2,2,3,3-tetrafluorooxetane with a halogenated hydrocarbon to obtain a mixture of 2,2,3,3-tetrafluoro-oxetane and the halogenated hydrocarbon containing a small amount of hydrogen fluoride.
2. The process according to claim 1, wherein the halogenated hydrocarbon is selected from the group consisting of trichloroethylene, tetrachloroethane, 1,1,1- and 1,1,2-trifluorotrichloroethane, 1,2-difluorotetrachloroethane, methylchloroform, tetrachloroethylene, p-chlorotoluene, p-chlorobenzotrifluoride, 3,4-dichlorobenzotri-fluoride and mixtures thereof.
3. The process according to claim 2, wherein the halogenated hydrocarbon is 1,1,2-trichloro-1,2,2-trifluoroethane or 1,2-difluorotetrachloroethane.
4. The process according to claim 3, wherein the halogenated hydrocarbon is 1,1,2-trichloro-1,2,2-trifluoroethane.
5. The process according to claim 1, which further comprises distilling the mixture of 2,2,3,3-tetrafluoro-oxetane and the halogenated hydrocarbon containing a small amount of hydrogen fluoride to obtain a mixture of 2,2,3,3-tetrafluorooxetane and the halogenated hydrocarbon containing a decreased amount of hydrogen fluoride and distilling

- 8 -

**0252454**

the mixture of 2,2,3,3-tetrafluoroacetone and the halogenated hydrocarbon to recover the mixture containing substantially no hydrogen fluoride.

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JP 61-97277A

## SPECIFICATION

## 1. TITLE OF THE INVENTION

## METHOD OF RECOVERING 2,2,3,3-TETRAFLUOROOXETANE

## 2. CLAIMS

(1) A method of recovering 2,2,3,3-tetrafluorooxetane, characterized by mixing a mixture of 2,2,3,3-tetrafluorooxetane and hydrogen fluoride with concentrated sulfuric acid at a temperature of not higher than 40°C, thereby separating the mixture into two layers, and then recovering the 2,2,3,3-tetrafluorooxetane.

(2) A method according to Claim 1, wherein the recovery is carried out by liquid-liquid separation.

(3) A method according to Claim 1, wherein a nitrogen gas is injected into the mixture as separated into two layers, thereby allowing the nitrogen gas to entrain and recover the 2,2,3,3-tetrafluorooxetane.

(4) A method according to Claim 1, wherein the 2,2,3,3-tetrafluorooxetane is recovered by subatmospheric distillation.

## 3. DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a method of recovering 2,2,3,3-tetrafluorooxetane, and more particularly to a method of recovering 2,2,3,3-tetrafluorooxetane from a mixture of 2,2,3,3-tetrafluorooxetane and hydrogen fluoride.

2,2,3,3,3-pentafluoropropanol can be prepared by reaction of tetrafluoroethylene, formaldehyde, and hydrogen fluoride. In the reaction, an excess portion of hydrogen fluoride works as a catalyst, and thus hydrogen fluoride is used in excess. Unreacted hydrogen fluoride contains by-product 2,2,3,3-tetrafluorooxetane, which is useful as a raw material for high-functional materials, and thus is desirable to recover and purify.

Hydrogen fluoride has a boiling point of 20°C, whereas 2,2,3,3-tetrafluorooxetane has a boiling point of 28°C, and thus it is quite difficult to separate one from another by distillation.

In view of these situations, the present inventors have made strenuous studies on separation of 2,2,3,3-tetrafluorooxetane from hydrogen fluoride, and have found that when the mixing of these two is carried out in the presence of concentrated sulfuric acid at a relatively low temperature, the 2,2,3,3-tetrafluorooxetane and the acid components are separated into two layers, respectively, and the 2,2,3,3-tetrafluorooxetane can be easily recovered therefrom, and have established the present invention.

That is, the object of the present invention is to provide a method of recovering 2,2,3,3-tetrafluorooxetane, characterized by mixing a mixture of 2,2,3,3-tetrafluorooxetane and hydrogen fluoride with concentrated sulfuric acid at a temperature of not higher than 40°C, thereby separating the mixture into two layers, and then recovering 2,2,3,3-tetrafluorooxetane therefrom.

Concentrated sulfuric acid can be used in an amount at least 5 times, preferably at least 8 times, as large as the weight of hydrogen fluoride.

Mixing of concentrated sulfuric acid can attain separation into two layers, i.e. an upper layer of 2,2,3,3-tetrafluorooxetane substantially free from acid components, and a lower layer of hydrogen fluoride-sulfuric acid adduct. These two layers can be separated one from another by liquid-liquid separation, or by injection of a nitrogen gas thereto to allow the nitrogen gas to entrain and recover 2,2,3,3-tetrafluorooxetane, or by subatmospheric distillation.

Hydrogen fluoride can be easily recovered from the acid adduct by the

ordinary means.

The present invention will be described in detail below, referring to Examples.

#### EXAMPLE 1

50g of 95wt.% sulfuric acid was charged into a polytrifluoroethylene pipe, 1/2 inch in diameter, provided with a stainless steel needle valve, and cooled with dry ice/methanol. Then, a mixture of 6.4g of hydrogen fluoride/10.3g of oxetane was introduced therein portion by portion, followed by tight closure, and by gentle mixing of sulfuric acid with hydrogen fluoride/oxetane. After there was no release of the heat of reaction by sulfuric acid and hydrogen fluoride, stirring was conducted vigorously for 10 minutes, followed by standing for one hour. Then, liquid-liquid separation was conducted.

The amount of the lower sulfuric acid layer recovered by liquid-liquid separation was 61.36g, whereas the amount of the remaining oxetane layer was 5.34g. The oxetane layer had a hydrogen fluoride concentration of 1.5wt.%, and the percent recovery was 51.1wt.%.

Then, the oxetane dissolved in the remaining sulfuric acid layer was stripped off by a nitrogen gas, allowed to bubble through water and collected through a 100-ml glass cold trap.

The conditions were nitrogen gas flow rate : 50ml/min., temperature : 20°C, and duration : one hour, whereby 1.2g of oxetane was collected. 0.13g of hydrogen fluoride was trapped in the water scrubber column (hydrogen fluoride concentration in oxetane was 9.8wt.%).

#### EXAMPLE 2

In the same apparatus and the same manner as in Example 1, a hydrogen fluoride/oxetane mixture (3.61g of hydrogen fluoride/1.97g of

oxetane) was mixed with 20.0g of sulfuric acid, and stripping by a nitrogen gas was conducted from the beginning without any liquid-liquid separation.

The conditions where nitrogen gas flow rate : 50ml/min., temperature : 0°C, and duration : 1.5 hours, whereby 1.96g of oxetane was recovered, and the hydrogen fluoride concentration was 0.18wt.%.

Applicant : Daikin Industries, Ltd.

Patent Attorney : T. Aoyama and other two.